In the Application of:

VINAY G. SAKHRANI, ET AL.	DOCKET NO.: 5764-001
APPLICATION NO.: 10/791,542	GROUP ART UNIT: 1773
FILED: MARCH 2, 2004	EXAMINER: RAMSEY ZACHARIA

FOR: ARTICLE WITH LUBRICATED SURFACE AND METHOD

DECLARATION OF VINAY G. SAKHRANI PURSUANT TO 37 C.F.R. § 1.132

I hereby declare as follows:

1. I am the same Vinay G. Sakhrani who is a named co-inventor of Application No. 10/791,542.

2. I am a graduate of North Carolina State University with a Masters of Science degree and a background in polymer materials, plasma science, and plasma assisted CVD coatings.

3. I am employed by TriboFilm Research, Inc. as Vice-President of Technology.

4. In the Office Action mailed on October 2, 2007 for the above-referenced application, the Examiner rejected all of the pending claims under 37 C.F.R. 103(a) as being unpatentable over U.S. Patent No. 4,822,632 (hereinafter Williams) in view of U.S. Patent No. 5,830,577 (hereinafter Murayama). The Examiner appears to base his rejection, in whole or in part, on the assertion that silicone oil and perfluoropolyethers are interchangeable with one another for use as lubricants based on the teachings of Murayama; that the perfluoropolyether of Murayama can be substituted for the silicone oil of Williams; and that the teaching of "any pressure" in relation to the pressure at which the plasma is generated encompasses the extreme vacuum conditions of Williams as well as the atmospheric pressure conditions of the above-referenced application. However, these assertions by the Examiner are incorrect.

To obtain a better appreciation for the differences between the present application and the teachings of Williams and Murayama, as well as the

unexpected results and advantages flowing therefrom, the Examiner is directed to paragraph 6 below.

5. The following experiments were carried out by me or under my direct supervision and control to demonstrate the advantages of the present application. Specifically, the following data demonstrate the advantageous properties obtained through the following differences in the teachings of Williams and Murayama and the teachings of the present application: the use of a perfluoropolyether lubricant exposed to an energy source, such as an ionizing gas plasma at atmospheric pressure, and not a silicone-based lubricant exposed to an ionizing gas plasma at extreme vacuum. Experiments A, C, and D are comparative experiments and do not represent embodiments of the present application. Experiments B and E are embodiments of the present application, and Experiments F and G include embodiments of the present application as well as comparative examples.

Comparative Experiment A

Syringes Prepared According to Example 2 of the Williams Patent

10cc polypropylene syringes barrels were coated with a 5 percent solvent solution of polydimethylsiloxane by dipping. The syringe barrels were allowed to dry at ambient conditions to evaporate the solvent. The syringe barrels were then exposed to an energy source, in this case a vacuum air plasma at the following conditions:

- Pressure in the plasma chamber – 300 millitorr
- Power – 125 watts
- Time of exposure to the plasma – 10 minutes

The syringe barrels were then assembled with unlubricated halobutyl rubber stoppers. The assembled syringes were then placed in a Harvard Apparatus syringe pump, and the force exerted by the syringe pump on the assembled syringe (that is, the force required to move the stopper in the syringe barrel) was measured using a Dillon AFG-100N digital force gauge. The travel speed was about 100 mm/min.

Figure 1 presents the results of this experiment. A very large force was required to initiate movement of the stopper (break-free force). The force required to maintain movement steadily increased until the force limit of the syringe pump was reached. This indicated that the lubricant was over-crosslinked causing a complete loss of its lubricating characteristics.

Experiment B

Syringes Prepared According to Applicants' Present Application

10 cc polypropylene syringe barrels were spray coated with a perfluoropolyether lubricant (Fomblin M100® from Solvay Solexis) and exposed to an energy source, in this case an ionizing gas plasma at about atmospheric pressure. The syringes were then assembled with unlubricated halobutyl rubber stoppers and tested on the same equipment used for Experiment A. Figure 2 (Experiment B) presents the results of this experiment and shows that only a very small break-free force was required to initiate movement, and that a minimal force was required to maintain movement (about 1/20th of the force required for the syringes treated according to the Williams teachings in Experiment A).

Comparative Experiment C

Comparison with Perfluoropolyether Lubricant Exposed to Vacuum Plasma

In order to demonstrate the difference between using plasma generated under conditions of extreme vacuum (Williams) as the energy source and plasma generated at about atmospheric pressure (present application) as the energy source, 10 cc polypropylene syringe barrels coated with the perfluorpolyether lubricant were exposed to the vacuum plasma according to the parameters of Experiment A (that is, the Williams patent). These syringe barrels were then assembled with unlubricated halobutyl rubber stoppers and tested with the same apparatus as described above. The results are shown in Figure 2 (Comparative Experiment C). While these syringes exhibited a low initial break-free force, the force needed to maintain movement steadily increased until the upper limit of the

force gauge was reached. This indicated that the perfluoropolyether lubricant was over-crosslinked causing a loss of its lubricating characteristics.

Comparative Experiment D

Comparison with Perfluoropolyether Lubricant Not Exposed to an Energy Source

As a comparative example, 10cc polypropylene syringe barrels were coated with the perfluoropolyether lubricant and were NOT exposed to an energy source. The lubricated barrels were then assembled with unlubricated halobutyl rubber stoppers and tested with the same apparatus as described above. The results are shown in Figure 2 (Comparative Experiment D). The syringes exhibited extreme stick-slip phenomenon where the stopper would alternately stick then suddenly move. This stick-slip phenomenon is due to lubricant migration from the barrel/stopper interface and is also observed in commercially available silicone lubricated syringes.

Experiment E

Plasma Treatment Prior to Application of the Lubricant

Uncoated 1ml glass syringe barrels were treated with a helium plasma at about atmospheric pressure according to the present application. The syringe barrels were then lubricated with Fomblin ZDOL followed by heating for 5 minutes to about 250°C. Fomblin ZDOL is a functionalized perfluoropolyether (PFPE) with hydroxyl (-OH) functional groups at both ends of the PFPE molecule.

The syringes were then assembled with halobutyl rubber stoppers and tested on the same equipment used for Experiment A. Figure 3 (Experiment E) presents the results of this experiment and shows that only a small break-free force was required to initiate movement, and minimal force was required to maintain movement without any stick-slip behavior.

Experiment F
Comparison of Perfluoropolyethers with and without Functional Groups (Binding Sites) and Plasma at Extreme Vacuum and Atmospheric Pressure

In order to demonstrate the difference between using plasma generated under conditions of extreme vacuum as the energy source and plasma generated at atmospheric pressure as the energy source, 12ml polypropylene syringe barrels were lubricated with a 50-50 mixture of Fomblin M (inert PFPE without functional end-groups) and Fomblin ZDOL (di-hydroxyl terminated PFPE, a lubricant with a binding site as in Murayama).

One set of syringe barrels was treated with a helium plasma at about atmospheric pressure according to the present application. The second set of syringe barrels was treated with an air plasma under vacuum conditions according to parameters of Experiment A (Example 2 of the Williams patent). Both set of syringes were assembled with unlubricated polyisoprene stoppers and tested with the same apparatus as described in Experiment A. Figure 4 presents the results of this experiment and shows that the syringes treated with atmospheric pressure plasma exhibited a low initial break-free force (about 1 pound of force) followed by minimal force to maintain movement without any stick-slip behavior. In comparison, syringes treated with vacuum plasma per Williams' teachings required about 8 pounds of force to initiate movement followed by continuously increasing force to maintain movement of the syringe stopper until the upper limit of the force gauge was reached. This indicated that the perfluoropolyether lubricant was over-crosslinked under vacuum plasma conditions causing a loss of its lubricating characteristics.

Experiment G
Comparison of Plasma at Extreme Vacuum and Atmospheric Pressure

In order to demonstrate the difference between using plasma generated under conditions of extreme vacuum as the energy source and plasma generated at atmospheric pressure as the energy source, 12ml polypropylene syringe barrels

were lubricated with Fomblin Y (inert branched PFPE without functional end-groups).

One set of syringe barrels was treated with a helium plasma at about atmospheric pressure according to the present application. The second set of syringe barrels was treated with an air plasma under vacuum conditions according to parameters of Experiment A (Example 2 of the Williams patent). Both set of syringes were assembled with unlubricated polyisoprene stoppers and tested with the same apparatus as described in Experiment A. Figure 5 presents the results of this experiment and shows that the syringes treated with atmospheric pressure plasma exhibited a low initial break-free force (about 2.2 pounds of force) followed by minimal force to maintain movement without any stick-slip behavior. In comparison, syringes treated with vacuum plasma per Williams' teachings required about 13 pounds of force to initiate movement followed by continuously increasing force to maintain movement of the syringe stopper until the upper limit of the force gauge was reached. This indicated that the perfluoropolyether lubricant was over-crosslinked under vacuum plasma conditions causing a loss of its lubricating characteristics.

Conclusions

- Beneficial results using the teachings of the Williams patent with silicone oil (Experiment A) could not be obtained. Results of Experiment A were similar to Experiment C in which the method of the Williams patent (plasma generated at extreme vacuum) was used with the lubricant of the present application.
- The poor results obtained when using the perfluoropolyether lubricant without any plasma treatment (Experiment D) indicates the importance of the atmospheric plasma treatment to achieve lubricity.
- Excellent results were achieved with the methods of the present application for both linear (Experiments B, C, and D) and branched (Experiment G) perfluoropolyethers without reactive functional groups.
- The results of Experiment F, in which a mixture of a lubricant without functional end groups (binding sites) and a lubricant with functional binding sites indicates that the lubricant of Murayama does not work with the teachings of Williams.

- The results of Experiment E indicate that pretreating the surface with ionizing gas plasma at about atmospheric pressure has beneficial results.

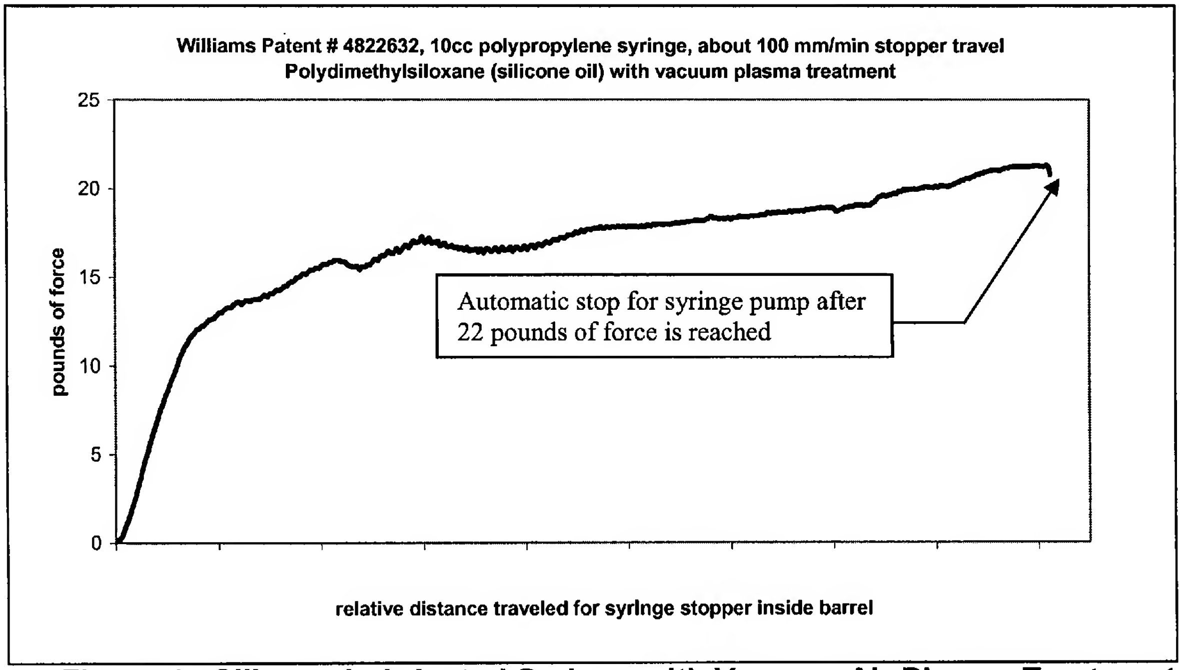

Figure 1. Silicone Lubricated Syringe with Vacuum Air Plasma Treatment According to Williams et al. (U.S. Patent No. 4,822,632)

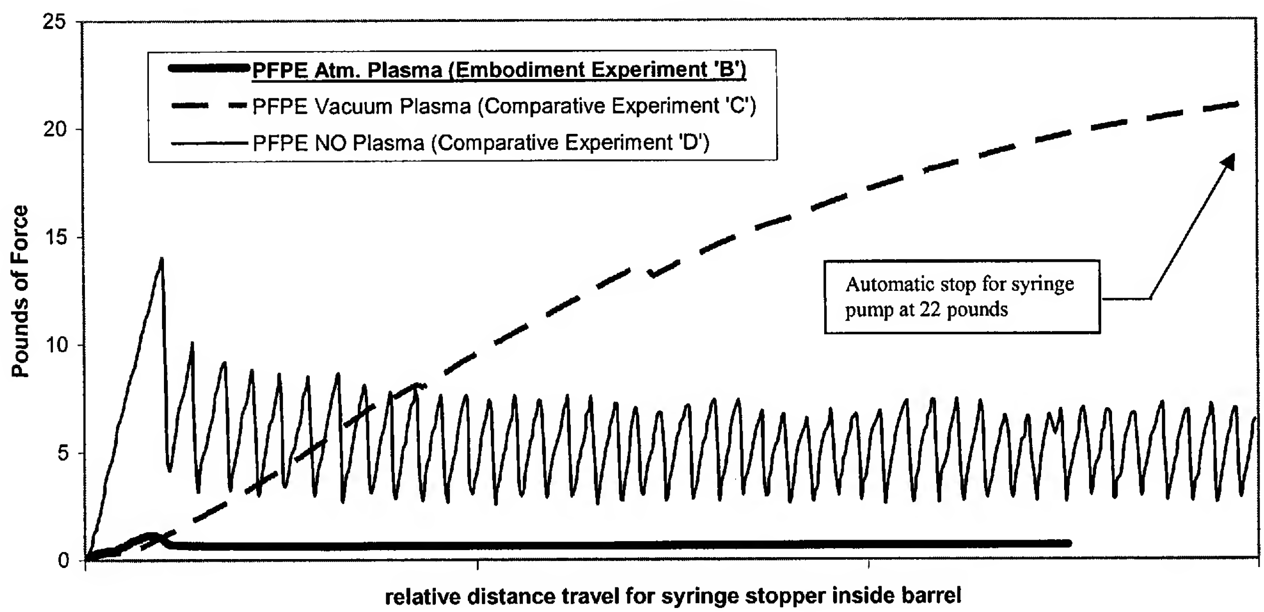

**Figure 2. Influence of Plasma Treatments on Perfluoropolyether (PFPE).
Embodiment "Experiment B" – PFPE with Atmospheric Plasma
Comparative Example "Experiment C" – PFPE with Vacuum Plasma
Comparative Example "Experiment D" – PFPE without Plasma Treatment**

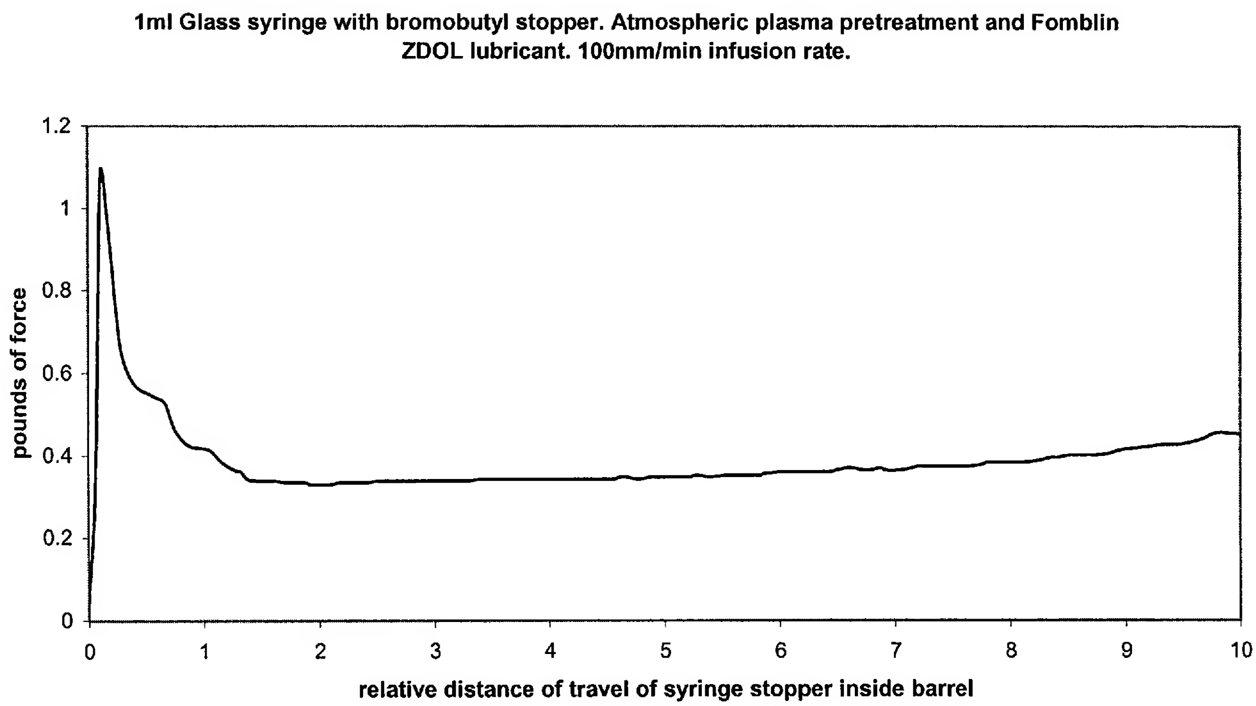

Figure 3. Fomblin ZDOL (di-hydroxyl terminated liner PFPE). Atmospheric plasma treatment only.

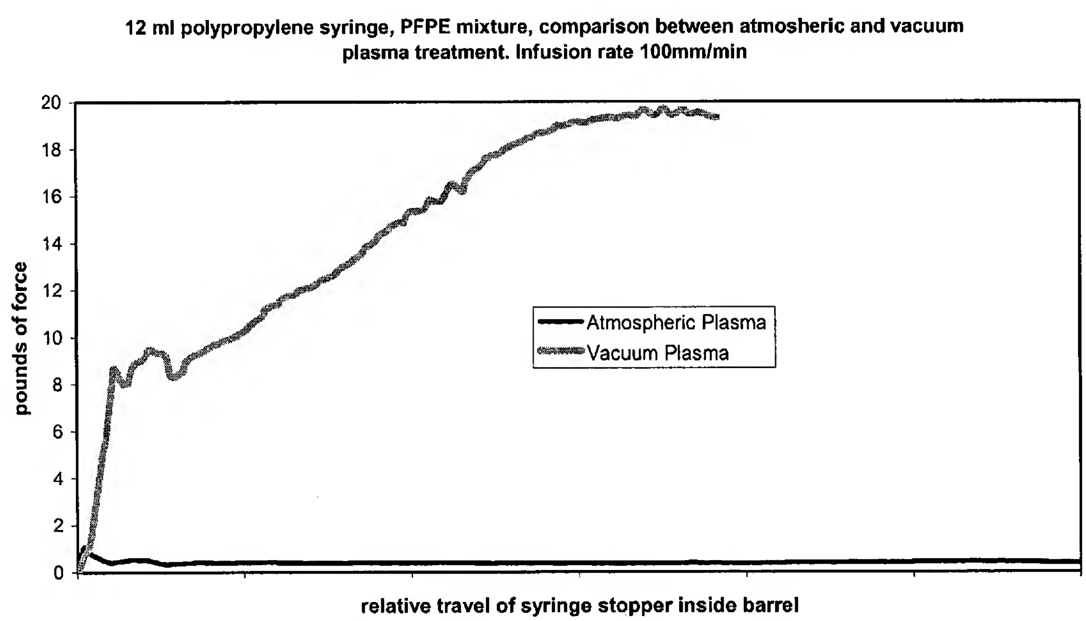

Figure 4: 50/50 mixture of Fomblin M 60 (inert linear PFPE) and Fomblin ZDOL (di-hydroxyl terminated liner PFPE). Atmospheric and Vacuum plasma comparison.

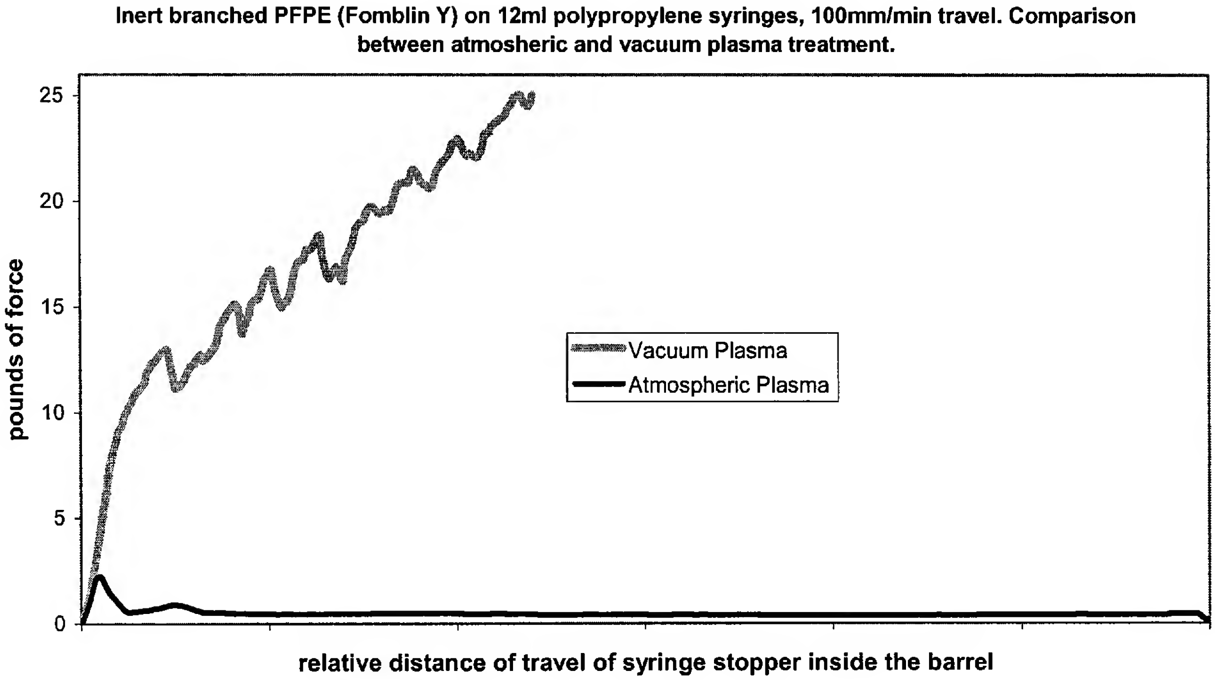

Figure 5: Inert branched PFPE lubricant Fomblin Y. Atmospheric and Vacuum plasma comparison.

6. Results

The results obtained from the above experiments prove that the Examiner's assertion that Williams and Murayama can be combined is incorrect. First, Williams does not produce a level of lubricity comparable to that obtained by the present application. Second, substituting the perfluoropolyether of the present application into the method of Williams produces poor results. If the teachings of Williams and Murayama are combinable as asserted by the Examiner, than the results of Experiment C should have been comparable to the results of Experiment B. Third, substituting a perfluoropolyether with binding sites into the method of Williams also produces poor results. Once again, if the teachings of Williams and Murayama are combinable, then the results of Experiment F for vacuum plasma should have been comparable to the results of Experiment B.

The difference in performance between the syringes prepared according to the Williams teachings and the present application provides unexpected and surprising results. It is clear from these results that the product obtained by the presently claimed method is significantly different than that produced according to the Williams teachings. The experimentation also shows that there is a significant difference between the effect produced by plasma generated under conditions of extreme vacuum and plasma generated at about atmospheric pressure. Finally, the experimentation showed that Murayama does not teach wholesale substitution of silicone oils and perfluoropolyethers without a host compound.

7. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing therefrom.

Vinay G. Sakhrani

December 27, 2007

Date